

[First Hit](#) [Fwd Refs](#) [Generate Collection](#)

L7: Entry 1 of 3

File: USPT

Apr 6, 2004

DOCUMENT-IDENTIFIER: US 6716274 B2

TITLE: Air filter assembly for filtering an air stream to remove particulate matter entrained in the stream

Brief Summary Text (4):

The invention also relates to polymer materials can be manufactured with improved environmental stability to heat, humidity, reactive materials and mechanical stress. Such materials can be used in the formation of fine fiber such as microfibers and nanofiber materials with improved stability and strength. As the size of fiber is reduced the survivability of the materials is increasingly more of a problem. Such fine fiber are useful in a variety of applications. In one application, filter structures can be prepared using this fine fiber technology. The invention relates to polymers, polymeric composition, fiber, filters, filter constructions, and methods of filtering. Applications of the invention particularly concern filtering of particles from fluid streams, for example from air streams and liquid (e.g. non-aqueous and aqueous) streams. The techniques described concern structures having one or more layers of fine fiber in the filter media. The compositions and fiber sizes are selected for a combination of properties and survivability.

Brief Summary Text (8):

The invention relates to polymeric compositions with improved properties that can be used in a variety of applications including the formation of fibers, microfibers, nanofibers, fiber webs, fibrous mats, permeable structures such as membranes, coatings or films. The polymeric materials of the invention are compositions that have physical properties that permit the polymeric material, in a variety of physical shapes or forms, to have resistance to the degradative effects of humidity, heat, air flow, chemicals and mechanical stress or impact.

Brief Summary Text (9):

In making non-woven fine fiber filter media, a variety of materials have been used including fiberglass, metal, ceramics and a wide range of polymeric compositions. A variety of techniques have been used for the manufacture of small diameter micro- and nanofibers. One method involves passing the material through a fine capillary or opening either as a melted material or in a solution that is subsequently evaporated. Fibers can also be formed by using "spinnerets" typical for the manufacture of synthetic fiber such as nylon. Electrostatic spinning is also known. Such techniques involve the use of a hypodermic needle, nozzle, capillary or movable emitter. These structures provide liquid solutions of the polymer that are then attracted to a collection zone by a high voltage electrostatic field. As the materials are pulled from the emitter and accelerate through the electrostatic zone, the fiber becomes very thin and can be formed in a fiber structure by solvent evaporation. As more demanding applications are envisioned for filtration media, significantly improved materials are required to withstand the rigors of high temperature 100.degree. F. to 250.degree. F. and up to 300.degree. F., high humidity 10% to 90% up to 100% RH, high flow rates of both gas and liquid, and filtering micron and submicron particulates (ranging from about 0.01 to over 10 microns) and removing both abrasive and non-abrasive and reactive and non-reactive particulate from the fluid stream.

h e b b g e e e f c e

e ge

Brief Summary Text (10):

Accordingly, a substantial need exists for polymeric materials, micro- and nanofiber materials and filter structures that provide improved properties for filtering streams with higher temperatures, higher humidities, high flow rates and said micron and submicron particulate materials. A variety of air filter or gas filter arrangements have been developed for particulate removal. However, in general, continued improvements are sought.

Brief Summary Text (12):

Herein, general techniques for the design and application of air filter arrangements are provided. The techniques include preferred filter media. In general, the preferred media concern utilization, within an air filter, of barrier media, typically pleated media, and fine fiber, to advantage. The filter media includes at least a micro- or nanofiber web layer in combination with a substrate material in a mechanically stable filter structure. These layers together provide excellent filtering, high particle capture, efficiency at minimum flow restriction when a fluid such as a gas or liquid passes through the filter media. The substrate can be positioned in the fluid stream upstream, downstream or in an internal layer. A variety of industries have directed substantial attention in recent years to the use of filtration media for filtration, i.e. the removal of unwanted particles from a fluid such as gas or liquid. The common filtration process removes particulate from fluids including an air stream or other gaseous stream or from a liquid stream such as a hydraulic fluid, lubricant oil, fuel, water stream or other fluids. Such filtration processes require the mechanical strength, chemical and physical stability of the microfiber and the substrate materials. The filter media can be exposed to a broad range of temperature conditions, humidity, mechanical vibration and shock and both reactive and non-reactive, abrasive or non-abrasive particulates entrained in the fluid flow. Further, the filtration media often require the self-cleaning ability of exposing the filter media to a reverse pressure pulse (a short reversal of fluid flow to remove surface coating of particulate) or other cleaning mechanism that can remove entrained particulate from the surface of the filter media. Such reverse cleaning can result in substantially improved (i.e.) reduced pressure drop after the pulse cleaning. Particle capture efficiency typically is not improved after pulse cleaning, however pulse cleaning will reduce pressure drop, saving energy for filtration operation. The filtration media can be cleaned using vibration-cleaning methods wherein the media is vibrated to loosen particulate that has collected on the surface. Such filters can be removed for service and cleaned in aqueous or non-aqueous cleaning compositions. Such media are often manufactured by spinning fine fiber and then forming an interlocking web of microfiber on a porous substrate. In the spinning process the fiber can form physical bonds between fibers to interlock the fiber mat into a integrated layer. Such a material can then be fabricated into the desired filter format such as cartridges, flat disks, canisters, panels, bags and pouches. Within such structures, the media can be substantially pleated, rolled or otherwise positioned on support structures.

Brief Summary Text (13):

The invention provides an improved polymeric material. This polymer has improved physical and chemical stability. The polymer fine fiber (0.0001 to 10 microns, 0.005 to 5 microns or 0.01 to 0.5 microns) can be fashioned into useful product formats. Nanofiber is a fiber with diameter less than 200 nanometer (about 0.2 micron). Microfiber is a fiber with diameter larger than 0.2 micron, but not larger than 10 microns. This fine fiber can be made in the form of an improved multi-layer microfiltration media structure. The fine fiber layers of the invention comprise a random distribution of fine fibers which can be bonded to form an interlocking net. Filtration performance is obtained largely as a result of the fine fiber barrier to the passage of particulate. Structural properties of stiffness, strength, pleatability are provided by the substrate to which the fine fiber adhered. The fine fiber interlocking networks have as important characteristics, fine fiber in the form of microfibers or nanofibers and relatively small spaces between the

fibers. Such spaces typically range, between fibers, of about 0.01 to about 25 microns or often about 0.1 to about 10 microns. The filter products comprising a fine fiber layer and a cellulosic layer are thin with a choice of appropriate substrate. The fine fiber adds less than a micron in thickness to the overall fine fiber plus substrate filter media. In service, the filters can stop incident particulate from passing through the fine fiber layer and can attain substantial surface loadings of trapped particles. The particles comprising dust or other incident particulates rapidly form a dust cake on the fine fiber surface and maintains high initial and overall efficiency of particulate removal. Even with relatively fine contaminants having a particle size of about 0.01 to about 1 micron, the filter media comprising the fine fiber has a very high dust capacity.

Brief Summary Text (14):

The polymer materials as disclosed herein have substantially improved resistance to the undesirable effects of heat, humidity, high flow rates, reverse pulse cleaning, operational abrasion, submicron particulates, cleaning of filters in use and other demanding conditions. The improved microfiber and nanofiber performance is a result of the improved character of the polymeric materials forming the microfiber or nanofiber. Further, the filter media of the invention using the improved polymeric materials of the invention provides a number of advantageous features including higher efficiency, lower flow restriction, high durability (stress related or environmentally related) in the presence of abrasive particulates and a smooth outer surface free of loose fibers or fibrils. The overall structure of the filter materials provides an overall thinner media allowing improved media area per unit volume, reduced velocity through the media, improved media efficiency and reduced flow restrictions.

Brief Summary Text (18):

Another preferred mode of the invention involves a single polymeric material combined with an additive composition to improve fiber lifetime or operational properties. The preferred polymers useful in this aspect of the invention include nylon polymers, polyvinylidene chloride polymers, polyvinylidene fluoride polymers, polyvinylalcohol polymers and, in particular, those listed materials when combined with strongly oleophobic and hydrophobic additives that can result in a microfiber or nanofiber with the additive materials formed in a coating on the fine fiber surface. Again, blends of similar polymers such as a blend of similar nylons, similar polyvinylchloride polymers, blends of polyvinylidene chloride polymers are useful in this invention. Further, polymeric blends or alloys of differing polymers are also contemplated by the invention. In this regard, compatible mixtures of polymers are useful in forming the microfiber materials of the invention. Additive composition such a fluoro-surfactant, a nonionic surfactant, low molecular weight resins (e.g.) tertiary butylphenol resin having a molecular weight of less than about 3000 can be used. The resin is characterized by oligomeric bonding between phenol nuclei in the absence of methylene bridging groups. The positions of the hydroxyl and the tertiary butyl group can be randomly positioned around the rings. Bonding between phenolic nuclei always occurs next to hydroxyl group, not randomly. Similarly, the polymeric material can be combined with an alcohol soluble non-linear polymerized resin formed from bis-phenol A. Such material is similar to the tertiary butylphenol resin described above in that it is formed using oligomeric bonds that directly connect aromatic ring to aromatic ring in the absence of any bridging groups such as alkylene or methylene groups.

Brief Summary Text (20):

An important aspect of the invention is the utility of such microfiber or nanofiber materials formed into a filter structure. In such a structure, the fine fiber materials of the invention are formed on and adhered to a filter substrate. Natural fiber and synthetic fiber substrates, like spun bonded fabrics, non-woven fabrics of synthetic fiber and non-wovens made from the blends of cellulosics, synthetic and glass fibers, non-woven and woven glass fabrics, plastic screen like materials both extruded and hole punched, UF and MF membranes of organic polymers can be

used. Sheet-like substrate or cellulosic non-woven web can then be formed into a filter structure that is placed in a fluid stream including an air stream or liquid stream for the purpose of removing suspended or entrained particulate from that stream. The shape and structure of the filter material is up to the design engineer. One important parameter of the filter elements after formation is its resistance to the effects of heat, humidity or both. One aspect of the filter media of the invention is a test of the ability of the filter media to survive immersion in warm water for a significant period of time. The immersion test can provide valuable information regarding the ability of the fine fiber to survive hot humid conditions and to survive the cleaning of the filter element in aqueous solutions that can contain substantial proportions of strong cleaning surfactants and strong alkalinity materials. Preferably, the fine fiber materials of the invention can survive immersion in hot water while retaining at least 50% of the fine fiber formed on the surface of the substrate. Retention of at least 50% of the fine fiber can maintain substantial fiber efficiency without loss of filtration capacity or increased back pressure. Most preferably retaining at least 75%.

Detailed Description Text (2):

The fine fibers that comprise the micro- or nanofiber containing layer of the invention can be fiber and can have a diameter of about 0.001 to 2 micron, preferably 0.05 to 0.5 micron. The thickness of the typical fine fiber filtration layer ranges from about 1 to 100 times the fiber diameter with a basis weight ranging from about 0.01 to 240 micrograms-cm.²

Detailed Description Text (9):

Addition polymers like polyvinylidene fluoride, syndiotactic polystyrene, copolymer of vinylidene fluoride and hexafluoropropylene, polyvinyl alcohol, polyvinyl acetate, amorphous addition polymers, such as poly(acrylonitrile) and its copolymers with acrylic acid and methacrylates, polystyrene, poly(vinyl chloride) and its various copolymers, poly(methyl methacrylate) and its various copolymers, can be solution spun with relative ease because they are soluble at low pressures and temperatures. However, highly crystalline polymer like polyethylene and polypropylene require high temperature, high pressure solvent if they are to be solution spun. Therefore, solution spinning of the polyethylene and polypropylene is very difficult. Electrostatic solution spinning is one method of making nanofibers and microfiber.

Detailed Description Text (26):

It should be understood that an extremely wide variety of fibrous filter media exist for different applications. The durable nanofibers and microfibers described in this invention can be added to any of the media. The fibers described in this invention can also be used to substitute for fiber components of these existing media giving the significant advantage of improved performance (improved efficiency and/or reduced pressure drop) due to their small diameter, while exhibiting greater durability.

Detailed Description Text (27):

Polymer nanofibers and microfibers are known, however their use has been very limited due to their fragility to mechanical stresses, and their susceptibility to chemical degradation due to their very high surface area to volume ratio. The fibers described in this invention address these limitations and will therefore be usable in a very wide variety of filtration, textile, membrane and other diverse applications.

Detailed Description Text (29):

The microfiber or nanofiber of the unit can be formed by the electrostatic spinning process. A suitable apparatus for forming the fiber is illustrated in FIG. 1. This apparatus includes a reservoir 80 in which the fine fiber forming polymer solution is contained, a pump 81 and a rotary type emitting device or emitter 40 to which the polymeric solution is pumped. The emitter 40 generally consists of a rotating

union 41, a rotating portion 42 including a plurality of offset holes 44 and a shaft 43 connecting the forward facing portion and the rotating union. The rotating union 41 provides for introduction of the polymer solution to the forward facing portion 42 through the hollow shaft 43. The holes 44 are spaced around the periphery of the forward facing portion 42. Alternatively, the rotating portion 42 can be immersed into a reservoir of polymer fed by reservoir 80 and pump 81. The rotating portion 42 then obtains polymer solution from the reservoir and as it rotates in the electrostatic field, a droplet of the solution is accelerated by the electrostatic field toward the collecting media 70 as discussed below.

Detailed Description Text (31):

In use, the polymer solution is pumped to the rotating union 41 or reservoir from reservoir 80. The forward facing portion 42 rotates while liquid exits from holes 44, or is picked up from a reservoir, and moves from the outer edge of the emitter toward collecting media 70 positioned on grid 60. Specifically, the electrostatic potential between grid 60 and the emitter 40 imparts a charge to the material which cause liquid to be emitted therefrom as thin fibers which are drawn toward grid 60 where they arrive and are collected on substrate 12 or an efficiency layer 14. In the case of the polymer in solution, solvent is evaporated off the fibers during their flight to the grid 60; therefore, the fibers arrive at the substrate 12 or efficiency layer 14. The fine fibers bond to the substrate fibers first encountered at the grid 60. Electrostatic field strength is selected to ensure that the polymer material as it is accelerated from the emitter to the collecting media 70, the acceleration is sufficient to render the material into a very thin microfiber or nanofiber structure. Increasing or slowing the advance rate of the collecting media can deposit more or less emitted fibers on the forming media, thereby allowing control of the thickness of each layer deposited thereon. The rotating portion 42 can have a variety of beneficial positions. The rotating portion 42 can be placed in a plane of rotation such that the plane is perpendicular to the surface of the collecting media 70 or positioned at any arbitrary angle. The rotating media can be positioned parallel to or slightly offset from parallel orientation.

Detailed Description Text (34):

The foregoing general description of the various aspects of the polymeric materials of the invention, the fine fiber materials of the invention including both microfibers and nanofibers and the construction of useful filter structures from the fine fiber materials of the invention provides an understanding of the general technological principles of the operation of the invention. The following specific exemplary materials are examples of materials that can be used in the formation of the fine fiber materials of the invention and the following materials disclose a best mode. The following exemplary materials were manufactured with the following characteristics and process conditions in mind. Electrospinning small diameter fiber less than 10 micron is obtained using an electrostatic force from a strong electric field acting as a pulling force to stretch a polymer jet into a very fine filament. A polymer melt can be used in the electrospinning process, however, fibers smaller than 1 micron are best made from polymer solution. As the polymer mass is drawn down to smaller diameter, solvent evaporates and contributes to the reduction of fiber size. Choice of solvent is critical for several reasons. If solvent dries too quickly, then fibers tends to be flat and large in diameter. If the solvent dries too slowly, solvent will redissolve the formed fibers. Therefore matching drying rate and fiber formation is critical. At high production rates, large quantities of exhaust air flow helps to prevent a flammable atmosphere, and to reduce the risk of fire. A solvent that is not combustible is helpful. In a production environment the processing equipment will require occasional cleaning. Safe low toxicity solvents minimize worker exposure to hazardous chemicals. Electrostatic spinning can be done at a flow rate of 1.5 ml/min per emitter, a target distance of 8 inches, an emitter voltage of 88 kV, an emitter rpm of 200 and a relative humidity of 45%.

Detailed Description Text (35):

h e b b g e e e f c e

e ge

The choice of polymer system is important for a given application. For pulse cleaning application, an extremely thin layer of microfiber can help to minimize pressure loss and provide an outer surface for particle capture and release. A thin layer of fibers of less than 2-micron diameter, preferably less than 0.3-micron diameter is preferred. Good adhesion between microfiber or nanofiber and substrates upon which the microfibers or nanofibers are deposited is important. When filters are made of composites of substrate and thin layer of micro- and nanofibers, such composite makes an excellent filter medium for self-cleaning application. Cleaning the surface by back pulsing repeatedly rejuvenates the filter medium. As a great force is exerted on the surface, fine fiber with poor adhesion to substrates can delaminate upon a back pulse that passes from the interior of a filter through a substrate to the micro fiber. Therefore, good cohesion between micro fibers and adhesion between substrate fibers and electrospun fibers is critical for successful use.

Detailed Description Text (36):

Products that meet the above requirements can be obtained using fibers made from different polymer materials. Small fibers with good adhesion properties can be made from such polymers like polyvinylidene chloride, poly vinyl alcohol and polymers and copolymers comprising various nylons such as nylon 6, nylon 4,6; nylon 6,6; nylon 6,10 and copolymers thereof. Excellent fibers can be made from PVDF, but to make sufficiently small fiber diameters requires chlorinated solvents. Nylon 6, Nylon 66 and Nylon 6,10 can be electrospun. But, solvents such as formic acid, m-cresol, tri-fluoro ethanol, hexafluoro isopropanol are either difficult to handle or very expensive. Preferred solvents include water, ethanol, isopropanol, acetone and N-methyl pyrrolidone due to their low toxicity. Polymers compatible with such solvent systems have been extensively evaluated. We have found that fibers made from PVC, PVDC, polystyrene, polyacrylonitrile, PMMA, PVDF require additional adhesion means to attain structural properties. We also found that when polymers are dissolved in water, ethanol, isopropanol, acetone, methanol and mixtures thereof and successfully made into fibers, they have excellent adhesion to the substrate, thereby making an excellent filter medium for self-cleaning application. Self-cleaning via back air pulse or twist is useful when filter medium is used for very high dust concentration. Fibers from alcohol soluble polyamides and poly(vinyl alcohol)s have been used successfully in such applications. Examples of alcohol soluble polyamides include Macromelt 6238, 6239, and 6900 from Henkel, Elvamide 8061 and 8063 from duPont and SVP 637 and 651 from Shakespeare Monofilament Company. Another group of alcohol soluble polyamide is type 8 nylon, alkoxy alkyl modifies nylon 66 (Ref. Page 447, Nylon Plastics handbook, Melvin Kohan ed. Hanser Publisher, New York, 1995). Examples of poly(vinyl alcohol) include PVA-217, 224 from Kuraray, Japan and Vinol 540 from Air Products and Chemical Company. We have found that filters can be exposed to extremes in environmental conditions. Filters in Saudi Arabian desert can be exposed to temperature as high as 150 F .degree. or higher. Filters installed in Indonesia or Gulf Coast of US can be exposed high humidity above 90% RH and high temperature of 100 F .degree.. Or, they can be exposed to rain. We have found that filters used under the hood of mobile equipment like cars, trucks, buses, tractors, and construction equipment can be exposed to high temperature (+200.degree. F.), high relative humidity and other chemical environment. We have developed test methods to evaluate survivability of microfiber systems under harsh conditions. Soaking the filter media samples in hot water (140 F .degree.) for 5 minutes or exposure to high humidity, high temperature and air flow.

Detailed Description Text (93):

Oligomers of para-tert-butyl phenol, molecular weight range 400 to 1100, was purchased from Enzymol International, Columbus, Ohio. These low molecular weight polymers are soluble in low alcohols, such as ethanol, isopropanol and butanol. These polymers were added to co-polyamide described earlier and electrospun into 0.2 micron fibers without adverse consequences. Some polymers and additives hinder the electrospinning process. Unlike the conventional phenolic resin described in

Example 2, we have found that this group of polymers does not interfere with fiber forming process.

Detailed Description Text (110):

We have prepared samples of mixture of Polymer A and Polymer B (Sample 6A) and another sample of mixture of Polymer A, Polymer B and Additive & (Sample 6B). We then formed fiber by electrospinning process, exposed the fiber mat at 300.degree. F. for 10 minutes and evaluated the surface composition by ESCA surface analysis.

Detailed Description Text (149):

Crosslinked, Electrospun PVA

Detailed Description Text (150):

PVA powders were purchased from Aldrich Chemicals. They were dissolved either in water or 50/50 mixture of methanol and water. They were mixed with crosslinking agent and toluene sulfonic acid catalyst before electrospinning. The resulting fiber mat was crosslinked in an oven at 150.degree. C. for 10 minutes before exposing to THC bench.

Detailed Description Text (160):

A conventional cellulose air filter media was used as the substrate. This substrate had a basis weight of 67 pounds per 3000 square feet, a Frazier permeability of 16 feet per minute at 0.5 inches of water pressure drop, a thickness of 0.012 inches, and a LEFS efficiency of 41.6%. A fine fiber layer of crosslinked PVA with polyacrylic acid of Example 12 was added to the surface using the process described with a nominal fiber diameter of 0.2 microns. The resulting composite had a LEFS efficiency of 92.9%. After exposure to 160F air at 100% relative humidity for 2 hours the substrate only sample was allowed to cool and dry, it then had a LEFS efficiency of 35.3%. After exposure to 160F air at 100% relative humidity for 2 hours the composite sample was allowed to cool and dry, it then had a LEFS efficiency of 83.1%. Using the mathematical formulas described, the fine fiber layer efficiency retained after 2 hours of exposure was 89%, the number of effective fine fibers retained was 76%.

Detailed Description Paragraph Table (15):

Sample 12A	Sample 12B	Sample 12C	Sample 12D	PVA	Hydrolysis	98-99	87-89	87-89	87-89
M.W. 31,500-	31,500-	31,500-	31,500-	50,000	50,000	50,000	50,000	PVA	10 10 10 10
Conc. (%)	Solvent	Water	Mixture	Mixture	(d)	Other	Polymer	None	None
Acrylic Acid	Cymel	385	Other	Polymer/	0	0	30	30	PVA (%)
(b)	20	(b)	Retained	THC,	1 hr.	%	Fiber	0	(a,b) 95
(a)	Retained	THC,	3 hr.	(a)	Retained	THC,	3 hr.	(a): Temperature	160.degree. F., 100% humidity
(b): Temperature	140.degree. F.,	100% humidity	(c): Molecular Weight	2000	(d): Melamine formaldehyde resin from Cytec				

Other Reference Publication (7):

Bognitzki, M. et al., "Submicrometer Shaped Polylactide Fibers by Electrospinning", Philipps-University Marburg, Department of Chemistry, Mainz, Germany, pp. 115 and 116 (Date unknown).

Other Reference Publication (8):

Chun, I. et al., "Carbon Nanofibers from Polyacrylonitrile and Mesophase Pitch" Jour. of Advanced Materials, vol. 31, No. 1, pp. 36-41 (Jan. 1999).

Other Reference Publication (9):

Deitzel, J. et al., "Generation of Polymer Nanofibers Through Electrospinning", Army Research Labm Aberdeen Proving Ground, MD 21005-5066, ARL-TR-1898, pp 1-36 (Jun. 1999).

Other Reference Publication (16):

Doshi, J., "The Electrospinning Process and Applications of Electrospun Fibers", UMI Dissertation Services University of Akron, pp. 1-170, including sections (Aug.

1994).

Other Reference Publication (17):

Fang, X., "Application of Electrospinning for Polymers", UMI Dissertation Services, University of Akron, pp. 1168, including sections (Aug. 1997).

Other Reference Publication (18):

Gibson, P. et al., "Electrospinning Technology: Direct Application of Tailorable Ultrathin Membranes", Jour. of Coated Fabrics, vol. 28, pp. 63-72 (Jul. 1998).

Other Reference Publication (23):

Kim, J. et al., "Polybenzimidazole Nanofiber Produced by Electrospinning", Polymer Engineering And Science vol. 39, No. 5, pp. 849-854 (May 1999).

h e b b g e e e f c e

e ge